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# MODERN ANALYTICAL TECHNIQUES FOR EVALUATING MIXED ENVIRONMENTAL EXPOSURES TO FIBROUS AND PARTICULATE DUSTS IN THE ASBESTOS INDUSTRY

National Center for Urban and Industrial Health
Cincinnati, Ohio

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# MODERN ANALYTICAL TECHNIQUES FOR EVALUATING MIXED ENVIRONMENTAL EXPOSURES TO FIBROUS AND PARTICULATE DUSTS IN THE ASBESTOS INDUSTRY

by

Robert G. Keenan and Richard E. Kupel*

## I Introduction

### A. Current Occupational Health Study of the Asbestos Products Industry in the United States

Although asbestosis has been recognized as an occupational disease since the early 1900's, related environmental exposure data are scanty or often unavailable from the prevalence or clinical studies made prior to the current PHS Study. The data from those early studies, even where available, are relevant only historically because changes within the industry as a result of technological advances have undoubtedly affected the levels and the nature of the dust to which workers are now exposed. The long latent period from the initial exposure to the onset of disease is an additional factor that has made the accumulation of data difficult.

To secure more definitive information on this disease process, the Occupational Health Program of the Public Health Service undertook a combined epidemiologic and environmental study of workers in the asbestos products manufacturing industry in the United States. This study is designed to provide more precise data on the health effects from exposure to the fibers and dusts of three commercially important asbestos minerals— chrysotile, amosite, and crocidolite— and on the nature and intensity of pertinent, mixed environmental exposures.

Since this study was initiated some 4 years ago, environmental data characterizing the nature and extent of in-plant exposures have been and are being obtained on each of five product areas—fiber preparation, carding, spinning, twisting, and weaving. Pertinent medical data are obtained through baseline and follow-up examinations on this population.

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16. Abstracts. Modern analytical methods, including sample treatment procedures and instrumentation, which are being used to characterize mixed environmental exposures in a current study of the asbestos industry in the U.S. are discussed. The presentation is limited to the relatively new techniques that are being used for this purpose. In addition, certain older techniques such as emission spectroscopy, electron microscopy, and absorption spectroscopy are also utilized.

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B. Types of Methods for Characterization of Asbestos and Other Etiologic Agents

Modern analytical methods and instrumentation used in the environmental phase of this study include atomic absorption spectrophotometry, emission spectroscopy, x-ray diffraction, electron microscopy, electron microprobe, spectrophotofluorometry, phase contrast and light microscopy, plus the necessary sample digestion and concentration procedures.

II. Types of Samples

A. Bulk Asbestos Materials

Bulk asbestos materials include the ores, the milled asbestos fibers, settled dusts, and finished products. Samples of these are being analyzed for specific types of asbestos, for metallic elements, for quartz and other forms of free silica, for other minerals, and for polycyclic hydrocarbons.

B. Airborne Fibers and Dusts

Airborne fibers and dusts are being collected by impinger and membrane filter sampling techniques. The impinger samples, taken with the midget impinger with 95% alcohol as the collecting medium, are counted as fibers and as total dust.[1] The membrane filter samples are subjected to a fiber count.[1] These two sampling and associated counting techniques are being carried out to permit us to compare our results with those reported previously by other investigators in this country and in Great Britain.

Membrane filter samples are also being collected in large numbers as personal samples in textile plant areas with essentially a pure chrysotile exposure. These are analyzed for magnesium and the results converted to express chrysotile exposures.

C. Settled Dusts

Rafter samples of settled dust are being collected by brush techniques. These are analyzed for asbestos minerals, metallic elements, and crystalline free silica.

D. Finished Products

The finished products sampled include asbestos yarn, brake linings, asbestos mats, and cloth, to mention only a few.

Samples of these are analyzed for asbestos minerals, polycyclic hydrocarbons, and metallic elements.

III. Treatment of Samples

A. Microsieving Technique

Our interest in separating respirable fibers and particulates from the coarser materials prompted us to experiment with sieving procedures for bulk and airborne materials. This interest increased upon our finding elevated concentrations of nickel and chromium in chrysotile that had been subjected to ball milling and hammer milling in chrome steel mills. Also, we found a buildup in the concentration of these heavy metals during the industrial milling of asbestos ore. Furthermore, we determined that there is a greater concentration of these metals in the finer dust fractions, a finding of hygienic significance.

Accordingly, we decided to conduct analyses of the metallic elements in the respirable fractions of the fiber and dust samples. In the dry sieving techniques, we encountered difficulty with sieve plugging. The problem was resolved by placing the sieve in an ultrasonic bath and suspending the asbestos in 95% ethyl alcohol within the sieve. With this technique we are able to separate fibers into those above and those below 10 microns. X-ray diffraction analysis of the sieved chrysotile has shown no change in the patterns of the sieved fractions as compared with that of the bulk material. The technique requires about 4 hours for an approximately 1-gram portion of the mineral, depending upon whether or not the material has been ground previously. During this period, sieved portions are removed at 15-minute intervals and more alcohol is added to the sieve. The sieve is electroetched nickel. Blank runs have demonstrated a negligible contribution of copper, nickel, chromium, cobalt, and manganese.

B. Digestion Procedures

It has been determined experimentally that a 20- to 30-minute digestion of chrysotile asbestos fibers with 5N hydrochloric or nitric acid is more than adequate to solubilize the metallic constituents of bulk and airborne samples containing this mineral. However, neither of these acids attacks amosite or crocidolite completely, and it is necessary to use hydrofluoric acid (HF) for the digestion of these types of asbestos prior to analysis of the metallic elements. These findings are presented in Table I.

In view of these findings, all samples for heavy metal analyses are now digested with HF in platinum dishes to insure a more complete solubilization of the metallic elements contained within the crystal lattice or between the fibrils of the asbestos material. The routine application of this digestion technique insures a more complete dissolution of all samples regardless of what we know or do not know about their mineral composition.

C. Column Chromatography

Cyclohexane extracts of airborne fibers and dusts collected on 8" X 10" fiber-glass filters or of bulk asbestos-containing materials are prepared by the method of Monkman.[2] The extraction is conducted overnight in a Soxhlet apparatus using a spectrofluorometric grade reagent. The 400 ml. extract is concentrated to 1 ml. and transferred to a silica gel column. (The silica gel is kept activated by storage in an oven at 150°C.) The aliphatic components are eluted with 400 ml. of iso-octane. The aromatic fraction is then eluted with 100 ml. of benzene. The polycyclic hydrocarbons present in the latter eluate are then separated by means of a column of activated 100- to 200-mesh alumina (150°C. in drying oven, followed by addition of water to provide 1.8% $H_2O$ in final alumina bed—if a high activity column of, for example, 1.4% $H_2O$ is used, the elution time is very long, and if >2% $H_2O$ is present, the elution is poor).[2] For this separation, the benzene extract is evaporated to dryness using filtered air and the residue is dissolved in a small volume of cyclohexane which is then transferred to the 10-cm.X 1.0-cm. (diam.) alumina column. After this column is capped with 0.5 cm. of deactivated alumina, the hydrocarbons are eluted in turn with cyclohexane. During these elutions, the presence of individual polycyclic hydrocarbons is established in the separate eluates by monitoring with a recording UV spectrophotometer. Addition of 1% ether after the decline of the eluted chrysene aids the subsequent elutions of benzo(e)pyrene, benzo(a)pyrene, and benzo(k)fluoranthene.

D. Sample Mounting for X-Ray Diffraction Analysis

The technique of mounting representative portions of dust samples on molecular membrane filters was described by Talvitie and Brewer.[3] A known weight of the finely comminuted sample is suspended in a 250-ml. MCA* volumetric flask with the aid of a wetting agent. This mixture is then dispersed

*Mention of commercial products does not imply indorsement by the Public Health Service.

by means of a small ultrasonic bath, and an aliquot of the thoroughly agitated suspension, containing a known fraction of the sample, is pipeted out and filtered through a molecular membrane filter. This technique provides a sample of known weight on the filter. Asbestos-containing dust samples are amenable to this procedure after a preliminary dry screening of gross particles through a 200-mesh sieve, followed by grinding to fiber lengths of <3.5 microns. The membrane filter containing the sample aliquot is mounted in the x-ray diffractometer.

Airborne dusts and fibers collected on a membrane filter are removed by placing the filter in a test tube containing a few drops of a suitable wetting agent and sufficient water to cover it. Immersion of the test tube in the ultrasonic bath for 5 minutes promotes the removal of the dust and fiber deposit from the filter. After the original filter from the test tube is removed, the re-suspended fibrous dust sample is mounted on a second membrane filter as described above.

E. Low-Temperature Dry Ashing

A commercial instrument is now available for the dry ashing of organic matter without volatilizing trace elements. This technique allows the oxidative reactions to proceed in a very gentle manner, thus avoiding any disturbance of the structure of crystalline substances and sintering of ultrafine particles. Thus, the technique appears to be promising for the isolation and non-alteration of mineral substances deposited in body tissues.

The principle of operation is based upon the production of "active" forms of oxygen when a commercial source of the latter is passed through a high-frequency electromagnetic field. These excited oxygen species attack the sample, which is maintained at about 1 mm. Hg pressure in an oxidation chamber from which the exhaust gases are removed by a mechanical vacuum pump. (Figure 1)

The glow region in the RF discharge of oxygen contains 10- to 20% of oxygen atoms and about an equivalent amount of electronically excited molecular oxygen[4] The rest of the plasma consists of normal ground-state oxygen molecules, some ionized species, and a free electron concentration of $1 \times 10^{11}$ $cm^{-3}$. A few millimeters away from the glow region, most of the ionic species have disappeared; the "active" species persist and are carried downstream before they deactivate reactively or by collision.

A concentration of oxygen atoms higher than expected results from predictions based upon the rates of the directly contributing electron-molecule reactions, molecule ion-electron reactions, and electronically excited molecular-oxygen dissociation. The high concentration of atomic oxygen is due in large part to catalytic effects of such foreign gases as hydrogen, nitrogen, or water vapor, which provide the kinetic pathways to atomic oxygen production.

We are using this low-temperature (approximately 150°C.), dry-ashing method experimentally to oxidize the organic matrix of lung and other biological tissues without changing the crystal lattice of deposited fibers and particulates.

## IV. Methods of Analysis

### A. Atomic Absorption Spectrophotometry

Atomic absorption spectrophotometry is a method of elemental analysis of samples in solution in an aqueous or organic solvent. A portion of this solution is aspirated into the flame (Figure 2) where the vaporized sample constituents are irradiated by a lamp containing a cathode made from the analysis element(s). The ground-state atoms of the element in the vaporized sample absorb the resonance-line radiation emitted by this same element in the hollow-cathode lamp, thereby effecting a decrease in the intensity of the light passing through the instrument's monochromator to the photodetector. The percentage of this absorption by the atomic vapor of the analysis element is a measure of the element's concentration in the sample. The advantages of the atomic absorption are twofold:

1. It provides a high degree of sensitivity, for a direct-reading method, because it detects elements at their ground-state energy levels. At temperatures of 3,000°K., and below, at which flames operate, more than 99.99% of the atoms of many elements are in their ground state.

2. It is highly specific, as it eliminates optical interference by operating on the very narrow resonance lines of the analysis element. (The resonance line is the transition involving a drop of an excited atom to its ground state in a single jump.)

We are using atomic absorption methods in our analysis of certain metallic elements present in samples of airborne asbestos fibers and dusts and samples of bulk asbestos minerals.[5]

In the case of fibrous dusts we measure the magnesium concentration in the personal samples collected on individual workers who are exposed to chrysotile, $3\,MgO \cdot 2\,SiO_2 \cdot 2H_2O$, and who are wearing membrane filter samplers. The filter samples are transferred to separate borosilicate beakers and digested with 5N HCl for 30 minutes. The samples are diluted to volume and analyzed directly. The magnesium 2852 A resonance line provides a sensitivity (1% absorption) of 0.01 µg. Mg per ml. of final solution, thus assuring ample sensitivity for this determination. In the case of samples where the use of this line requires too great a dilution, the magnesium 2026 A line is used. In either event, the estimated magnesium concentration is converted to a calculated quantity of chrysotile in the total filter sample.

Comparison of this method with the x-ray diffraction procedure has shown a variation of results no greater than ± 10- to 15% for a limited number of samples containing a sufficiently large dust deposit for the x-ray technique. The atomic absorption method for magnesium has permitted us to run thousands of personal samples, including many whose total dust weighed less than 0.1 mg. The application, of course, is limited to exposures containing only one source material of the analysis element, namely, chrysotile asbestos in this case. However, where only one etiologic agent containing the analysis element is present in an airborne dust, the technique offers the recognized advantages of high sensitivity, accuracy, specificity, and speed common to all atomic absorption methods. The error of the method is ≤± 5% as determined from the analysis of known samples in our laboratory.

We are also using atomic absorption to determine numerous heavy metals in bulk samples of chrysotile, amosite, and crocidolite, the asbestos minerals used to the greatest extent in this country[5]. For this purpose, 0.25 g. of a bulk material is digested with hydrofluoric acid in a platinum dish for 3 to 4 hours, a procedure which involves a double evaporation to dryness. The final residue is dissolved in 1:1 redistilled hydrochloric acid and double distilled water and then filtered through a Whatman No. 42 paper. After filter is washed, the solution is concentrated and then finally diluted to 25 ml. A one-half portion of this solution is reserved; the remainder is diluted, as required, for the individual analyses. Ammonium chloride is added to provide a concentration of 20 mg./ml. to reduce the interference of iron with chromium (iron suppresses the chromium response—200 µg·Fe will suppress the response of 5 µg. Cr to that of <3 µg.Cr). The elements we are determining routinely in asbestos samples

are listed in Table II along with their sensitivities (amounts giving 1% absorption) using the stated resonance lines.

Use of the atomic absorption method for the stated analyses has provided us with results that are more specific, more sensitive, more accurate, and much more rapidly obtainable than those by other methods, including emission spectroscopy. Furthermore, it provides the great advantage over emission spectroscopy of being almost completely free of matrix effects, which is a tremendous asset in shifting from one type of sample to another as we are doing in our study of the asbestos industry.

B. X-Ray Diffractometry

X-rays constitute the 0.5 to 200 Angstrom region of the electromagnetic spectrum. Produced by electron bombardment of a heavy metal target, such as copper, cobalt, chromium, iron, or molybdenum, they are used in the laboratory for the study of crystal structure, measurement of stress in solids, phase equilibria, chemical analysis, and other diverse problems.

In occupational health laboratories, we use x-rays for chemical analysis. The x-ray diffractometer is an analytical instrument designed to measure directly the intensity of a beam of x-rays diffracted by a plane of atoms in a crystalline material. Thus, this technique is extremely useful for both qualitative and quantitative analysis of the crystalline compounds present in such materials as industrial dusts, ores, chemical intermediates, and finished products whose composition is of interest to the industrial hygienist in defining the chemical nature of the substances to which the worker is exposed.

We also use this method for the quantitative determination of the different crystalline forms of free silica in the presence of one another.[6] The chemical method does not distinguish between quartz, cristobalite, and tridymite.

Another example of its usefulness is the analysis of milligram quantities of chrysotile, amosite, and crocidolite—the three most commonly used forms of asbestos. Prior to our present study of this industry, evaluation of the environmental exposure of the worker was limited to the counting of fibers and particulates in samples collected by impingement or by filtration techniques.[1] Since initiating the present study, we have developed both external[7] and internal[8,9] standard methods for the x-ray diffraction analysis of asbestos minerals. The samples are ground dry in a mixer mill to a fiber length

of <3.5μ and passed through a 325-mesh U.S. Standard sieve prior to mixing with a prescribed quantity of the internal standard. Standard samples of the pure asbestos mineral and the internal standard, prepared in a series of known weight ratios, are used to calibrate the method. Quartz serves as the internal standard for amosite and crocidolite and aquamarine for chrysotile. The ratios of the intensities of the quartz 3.34 A peak to the crocidolite 3.11 A peak (or amosite 3.08 A) are plotted against the weight ratios to obtain the standard curve. With aquamarine the 3.28 A peak is used as the internal standard.

We use this method for the quantitative determination of each of the three asbestos minerals in bulk or settled dust samples. Recoveries over the 1 to 10 mg. range extends from 85 to 103%.

C. Electron Microprobe

The electron microprobe is an instrument (Figure 3) for spot chemical analysis by means of a finely-focused electron beam to excite characteristic x-radiation from a small volume at the surface of a solid specimen. (Figure 4). The wavelength and intensity of the characteristic x-ray lines are then used to identify the elements that are present at the target site and their relative mass concentrations. At the present time, analyses can be performed for all elements of atomic number 5 and greater.

The commercial electron microprobe usually consists of up to three curved crystal x-ray monochromators that are used to analyze the characteristic x-ray lines, which have been excited by the finely-focused electron beam, impinging upon a selected spot on the surface of the sample material. (Figure 5). The sample composition is determined from the wavelength and the intensity of these characteristic x-ray lines. The specimen may be translated under the electron beam for analysis of specific areas, which are located with the aid of an optical microscopic viewing system.

The instrument is capable of producing an electron focal spot of the order of 0.5 micron in diameter depending on the beam power and the sample being analyzed. The effective spot obtained with the electron beam is smaller when a material of high atomic numbers is analyzed than when a material of low atomic numbers is analyzed because of the additional penetration of the beam into the low-atomic sample. The use of a low intensity beam will permit the analysis of biological and asbestos type samples. The electron microprobe can be used to identify metallic impurities located on the surfaces of or associated within the lattice of asbestos fibers.

Fibrous bodies in lung tissues can be identified and analyzed with the electron microprobe.

Filter samples can be analyzed to determine if metallic impurities are associated with the fibers themselves or are located in the fines collected with the fibers.

Investigation has been initiated, as part of our current study, using the electron microprobe to determine the presence or absence of nickel, chromium, cobalt, and manganese associated with airborne asbestos samples. For the samples analyzed to date we have observed that these elements do not appear to be present in the asbestos fibers, but only in the associated dusts. The sensitivity of this technique, under ideal conditions, is shown in Table III.

D. Neutron Activation Analysis

This technique is based upon the interaction of neutrons with the stable nuclei of chemical elements. These nuclear reactions are similar to ordinary chemical reactions in that they exhibit a mass change and an energy of activation and they proceed at definite reaction rates, which depend upon the experimental conditions. There are several sources of neutron particles available, but research reactors, with their high thermal neutron flux, are generally used to irradiate samples for trace element analyses.

In our present study, we have had the opportunity to compare the results of neutron activation analysis with those obtained by atomic absorption spectrophotometry as applied to duplicate portions of a chrysotile sample. These comparisons are given in Table IV.

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## V. SUMMARY

Modern analytical methods, including sample treatment procedures and instrumentation, which are being used to characterize mixed environmental exposures in a current study of the asbestos industry in this country are discussed. The presentation is limited to the relatively new techniques that are being used for this purpose. In addition, certain older techniques such as emission spectroscopy, electron microscopy, and absorption spectroscopy are also utilized in this study.

Table I

Evaluation of Acid Digestion Methods for 0.5-Gram Portions of Asbestos Minerals

| Type of Asbestos | Element | Digestion Procedure | |
|---|---|---|---|
| | | HCl | HF |
| | | (µg. element/g. sample) | |
| Chrysotile | Ni | 365 | 380 |
| | Cr | 197 | 220 |
| | Co | 37 | 30 |
| | Mn | 270 | 270 |
| Amosite | Ni | None | 50 |
| | Cr | 20 | 35 |
| | Co | 5 | 18 |
| | Mn | 910 | 4,450 |
| Crocidolite | Ni | None | 20 |
| | Cr | 10 | 25 |
| | Co | None | 10 |
| | Mn | 55 | 180 |

Table II

Elements Determined by Atomic Absorption Spectrophotometry Applied to HF Digests of Asbestos Minerals

| Element | Resonance Line, A | Sensitivity* (1% Absorption) 1 X Scale | 3 X Scale |
|---|---|---|---|
| Co | 2407 | - | 0.2 |
| Cr | 3579 | 0.4 | 0.15 |
| Fe | 3720 | 1.0 | - |
| Fe | 2483 | 0.15 | - |
| Mn | 2795 | 0.1 | - |
| Ni | 2320 | 0.4 | 0.15 |

*Determined in authors' laboratory; expressed as µg./ml.

Table III

Sensitivity of Electron Microprobe Analyzer

| Element | Min. Detect. Mass, g. | Min. Detect. % in 1μ Particle | Spectrometer |
|---|---|---|---|
| Be | $1 \times 10^{-14}$ | 20. | Grating |
| Ca | $8 \times 10^{-16}$ | 0.1 | ADP-LiF |
| Co | $4 \times 10^{-16}$ | 0.03 | LiF |
| Cr | $5 \times 10^{-16}$ | 0.03 | LiF |
| Fe | $4 \times 10^{-16}$ | 0.03 | LiF |
| Mn | $5 \times 10^{-16}$ | 0.03 | LiF |
| Ni | $2 \times 10^{-16}$ | 0.04 | LiF |
| Pb | $5 \times 10^{-14}$ | 0.01 | LiF-ADP |

Table IV

Micrograms Element/Gram Asbestos

| Element | Atomic Absorption Method | Neutron Activation Analysis Method |
|---|---|---|
| Ni | 1690 | 1400 |
| Cr | 1090 | 1060 |
| Co | 78 | 80 |
| Mn | 423* | 1100 |
| | 538* | |

*Separate acid leachings.

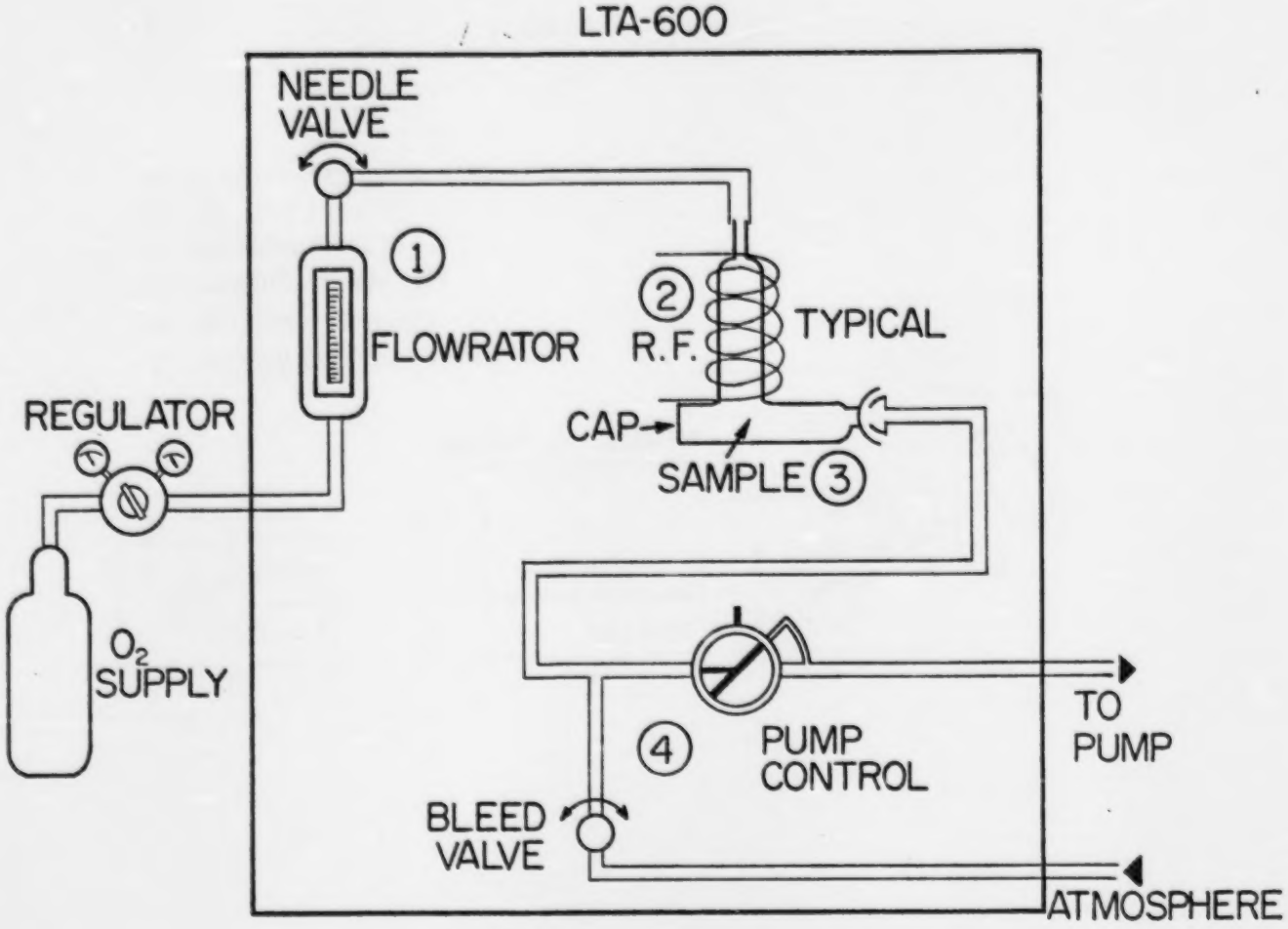
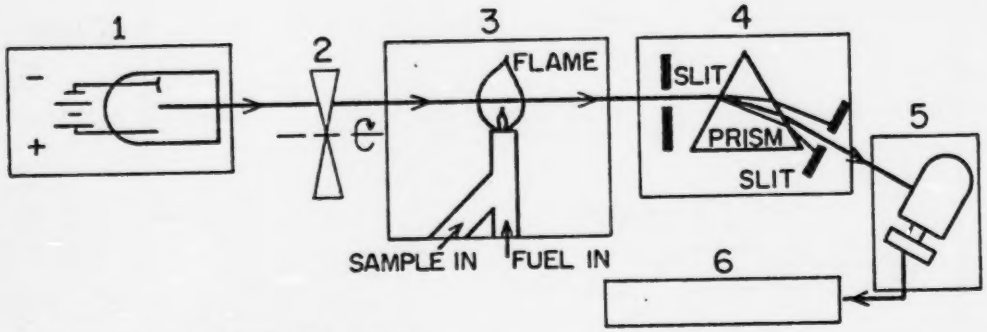

LTA-600
NEEDLE VALVE
1
FLOWRATOR
2
R.F.
TYPICAL
REGULATOR
CAP
SAMPLE
3
$O_2$ SUPPLY
4
PUMP CONTROL
TO PUMP
BLEED VALVE
ATMOSPHERE

Figure 1.




1. HOLLOW-CATHODE LAMP
2. ROTATING CHOPPER
3. VAPORIZING SYSTEM
4. MONOCHROMATOR
5. PHOTODETECTOR
6. ELECTRONICS AND READOUT

Figure 2.

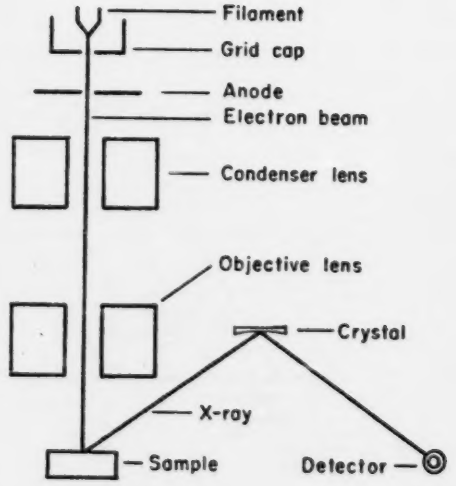

Filament
Grid cap
Anode
Electron beam
Condenser lens
Objective lens
Crystal
X-ray
Sample
Detector

Figure 3.

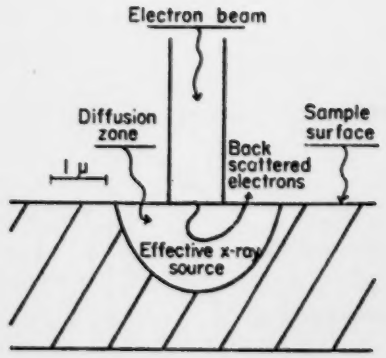

Electron beam
Diffusion zone
Sample surface
1 μ
Back scattered electrons
Effective x-ray source

Figure 4.

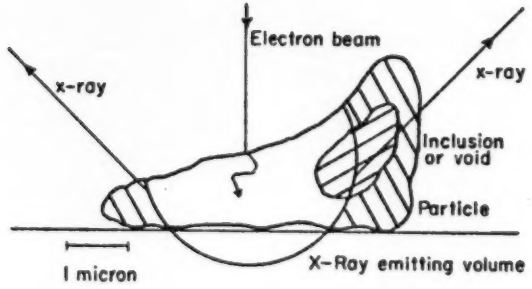

Electron beam
x-ray
x-ray
Inclusion
or void
Particle
1 micron
X-Ray emitting volume

Figure 5.